

Low-Velocity, Multi-Impact Durability Performance of Thick-Section 3WEAVE S2-Glass/SC-15 Composites Toughened With Thermoplastic Polyurethane Inter-Layer Films

by Steven E. Boyd, Ryan P. Emerson, and Travis A. Bogetti

ARL-TR-6547 August 2013

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14. ABSTRACT

For the past decade, the U.S. Army has invested a considerable amount of effort into assessing the suitability of plain-weave S2-glass fabric/SC-15 epoxy composites to replace conventional, monolithic materials (e.g., metals) in Army structural and ballistic applications. One performance-limiting material response repeatedly encountered with this composite system is interlaminar delamination under both ballistic and low-velocity impact (LVI). Strategies for improving the delamination resistance of thick-section composites are currently being developed and include the use of thicker composite plies combined with compliant films in the inter-ply region. Specifically, the LVI performance of 3WEAVE S2-glass/SC-15 composites toughened with thermoplastic urethane film inter-layers of various thicknesses are being investigated. This report documents the significant improvements in delamination resistance and durability performance that have been realized by following this strategy.

15. SUBJECT TERMS

thick-section composite, fabric composite, durability, damage tolerance, low-velocity impact, thermoplastic

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1. Introduction

Composites offer many advantages over conventional, monolithic materials (e.g., metals). Higher specific strength and stiffness make composites extremely attractive material options for a large number of applications. The U.S. Army has invested a considerable amount of effort into assessing the suitability of thick-section (>25-mm-thick) laminated plain weave (PW) S2-glass fabric/SC-15 epoxy composites as a viable replacement to conventional metals in Army structural and ballistic applications. Interlaminar delamination remains the primary performance-limiting behavior of this composite system, ultimately translating into reduced damage tolerance and durability. The areal density of one ply of PW S2-glass fabric/SC-15 epoxy composite is roughly 744 g/m² (or 24 oz./yd²) and a 1-in-thick laminate of this material requires 44 individual plies. Since delaminations occur at the resin-rich interfaces between the individual plies or layers, 43 potential delamination sites exist in every inch thickness of laminate for this composite system.

The focus of this report is the exploration of inter-ply hybridization to improve the delamination resistance of such thick-section composites. The strategy is to use thicker composite plies or so-called "sub-layers" with tough thermoplastic polyurethane (TPU) films in the inter-ply regions. This approach essentially reduces the number of potential delamination sites for any given areal density of laminate and toughens the remaining sites.

The Boeing Compression After Impact Test (1, 2) is a well-established method for characterizing the durability and damage tolerance of thin (<5-mm-thick) aerospace composites subjected to low-velocity single impact (e.g., tool drop, bird strike). This method has been used for decades by the U.S. Air Force with great success for developing and ranking new materials for highperformance aircraft applications (e.g., stiffness critical thin-sectioned wing skins with highdamage tolerance requirements). A novel material evaluation protocol employing multiple successive out-of-plane impacts has been recently developed (3–5) to assess the durability and damage tolerance of thick-section composites for Army applications. This protocol is used in the present research to rank the durability performance of composite panels under low-velocity impact (LVI) loading. Successive multiple out-of-plane impact on thick-section composite laminates represents a unique loading environment for many of the Army's structural and ballistic composite applications, including ground and air vehicle platforms. This load spectrum is not typically encountered or addressed by other branches of the Department of Defense (e.g., Air Force, Marines, or Navy)—or even the commercial sector. Compared to single event impact, the response of composite laminates subjected to multiple impacts has received relatively little attention in the literature (6-8). Most of the cited literature for multiple impacts deals with thinsectioned composite laminates rather than thick-sectioned composites (9-17).

2. Experimental

2.1 Textiles and Film Inter-Layers

Two types of textiles and two thicknesses of TPU film are used to fabricate the panels that were tested in this investigation. The two-dimensional (2-D) textile material is a plain weave S2-glass fabric with an areal density of 813 g/m² (24 oz/yd²) (18). The 3-D glass textile material is manufactured by 3Tex corporation (19) and consists of non-crimped in-plane S2-glass reinforcement with out-of-plane S2-glass stitching, having an overall areal density of 3505 g/m² (103.4 oz./yd²).

The TPU material is a single-layer solvent-free thermoplastic ester-based polyurethane, sold as a film adhesive product UAF-472 by Adhesive Films, Inc. (20). Two thicknesses of this film were used as inter-layers in this investigation, 0.13- and 0.25-mm (0.005- and 0.010-in) thick.

2.2 Processing of Impact Panels

The 2-D reinforced composites were processed and constituent materials were supplied by the U.S. Army Research Laboratory (ARL). The S2-glass fabric lay-up was quasi-isotropic, [(45/0)₅/45]_S, consisting of 22 plies (layers of fabric) and was infused with SC-15 resin (a two-phase toughened epoxy manufactured by Applied Poleramic, Inc. [21]). The infusion was performed using the vacuum-assisted resin transfer molding (VARTM) process. Large panels (approximately 89-cm squares) were processed, cured, and post-cured according to manufacturers' recommendations. Four 40.64-cm square impact samples were cut from the large panel using a water jet.

All 3-D reinforced composites were processed and constituent materials were supplied by the Center for Composite Materials (CCM) at the University of Delaware. The 3-D S2-glass preforms were cut oversize and stacked to five layers with the warp and weft directions of each layer aligned. The stacked layers were bagged between caul plates and infused with SC-15 using the VARTM method. Each sample was processed individually. After infusion, the resin was allowed to cure for 48 h at room temperature with a post-cure at 200° F for 4 h. After post-cure, individual samples were cut to 40.64-cm square dimension. If

the sample contained TPU film inter-layers, the TPU inter-layers were removed from their paper release liner and inserted ("placed") between each of the 3-D pre-forms in the lay-up and infusion and post-cure carried out as discussed; no infusion issues such dry patches, voids, and "race-tracking" were encountered with the 3-D pre-form lay-ups. Only one composite sample was processed for each panel type containing 3-D reinforcement due to limited stock and availability of all constituent materials. Table 1 shows details of the constituents, sample geometries, and densities.

Table 1. Material constituents, sample geometries, and densities.

Composite (SC-15 Resin)	Configuration and Lay-Up		Boundary Condition	Thickness (cm)	Weight (g)	Areal Density kg/m² (lbs/ft²)
2-D 24 oz S2/SC-15 Quasi		22 ply plain weave S2-glass, quasi- isotropic [(45,0) ₅ /45] _S	Clamped	1.51 ± 0.01	4370 ± 51	$26.6 \pm 0.18 \\ (5.46 \pm 0.04)$
3WEAVE S2/SC- 15 (no TPU)		5 × 3WEAVE S2-glass	Clamped	1.42	4217	25.2 (5.17)
3WEAVE S2/SC- 15 + 5 mil TPU		5 × 3WEAVE S2-glass + 0.005 in TPU inter-layers	Clamped	1.47	4400	26.4 (5.41)
3WEAVE S2/SC- 15 + 10 mil		5 × 3WEAVE S2-glass + 0.010 in TPU inter-layers	Clamped	1.60	4621	27.9 (5.7)

2.3 Drop Tower

An Instron Dynatup model 8110 impact test system was used for drop tower LVI experiments. The impactor was a 50-cm hemispherical tup, and the strain-gage based load cell had a capacity of 222.4 kN (50 kips). Force data during each impact were recorded for 25 ms duration at a sampling rate of 270 kHz. Tup displacement was calculated by converting the force to tup acceleration and numerically integrating twice with respect to time.

2.4 Impact Table

Impact testing was performed on a rigid stand with a thick steel frame that clamped around the perimeter of the sample. The entire stand-frame assembly was secured with 16 bolts that were tightened with a pneumatic wrench to minimize rigid-body motion of the sample after impact and rebound. Figure 1 illustrates the overall panel dimensions and the area of the outside perimeter that was under a clamped constraint.

2.5 Impact Location and Energy

Figure 1 also shows the locations of impact for all samples in this investigation. Impacts were conducted one-at-a-time to permit non-destructive evaluation of each panel after each impact. Mass and height of the impactor were 227.4 kg and 96 cm, respectively, yielding an impact energy of 2141 J and velocity of 4.34 m/s.

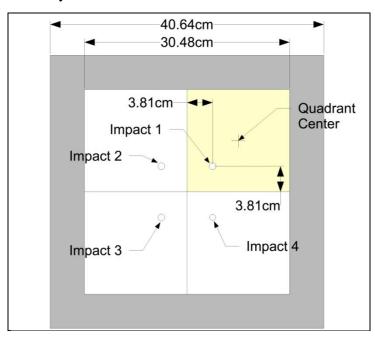


Figure 1. Illustration of clamped area of panel and impact location.

2.6 Non-Destructive Evaluation

The samples were removed from the impact table and ultrasonically c-scanned before and after each impact with a transducer frequency of 0.5 MHz and x-y resolution of 1 mm. These scans were used to track the evolution of damage in the sample. Delamination was observed as the predominant damage mode in the samples. All scans reported in this work were performed with the scanner operating in through-transmission mode. This approach provided an accurate determination of the extent of delamination across the full dimension of the samples.

3. Results

3.1 Force Versus Displacement

Force-deflection curves are given for all samples in figures 2–5. The force-deflection curves of figure 2 are representative of the set of four samples tested for quasi-isotropic 2-D S2-glass composites. The quasi-isotropic lay-up represents the baseline material system as it is the most characterized and will serve as the point of comparison for the key impact metrics discussed in this report. The force-deflection curves per impact shown in figure 2 illustrate a quantifiable degradation in material stiffness as shown by the decreasing slopes of the impact event portion of the curve. The peak loads on each impact are similar indicating that no one particular impact caused excessive damage (localized crushing under the impactor and shear deformation) to the sample. The rebound portions of the curves also show increasing deflections and permanent out-of-plane deformations with successive impacts. In summary, this material system displays significant quantifiable damage after four impacts and yields useful data for calculating a stiffness of the sample during impact and a progressive reduction in stiffness (herein referred to as the "degradation rate") between impacts.

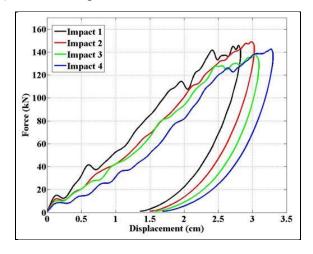


Figure 2. Force versus displacement for 2-D S2/SC-15.

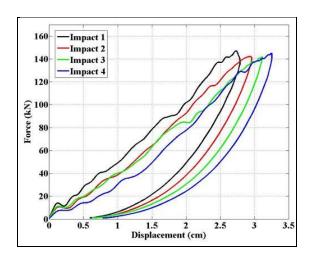


Figure 3. Force versus displacement for 3WEAVE S2/SC-15 (no TPU).

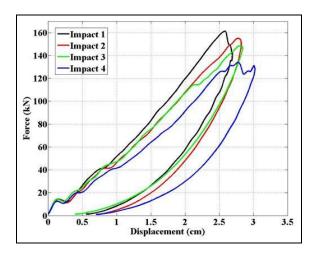


Figure 4. Force versus displacement for 3WEAVE S2/SC-15 + 5 mil TPU.

The 3WEAVE* S2/SC-15 composite response in figure 3 displays force deflection behavior that is very similar to the 2-D S2/SC-15 material. One difference in behavior is that the 3-D material sustains a lower permanent deflection of 0.5 cm to 1 cm. It is interesting that the peak deflections are approximately equal given the different fiber architectures present in the 2-D woven roving (undulating) and the 3-D pre-form (unidirectional with z-direction warp weaver). The difference in fiber architectures may explain the difference in permanent deformation between the materials. Another important observation is that reducing the number of plies (and, hence, delamination sites) alone does not improve the degradation rate of the material. The 22-ply 2-D S2/SC-15 material has 21 interfaces between fiber layers while the 3-D pre-form only has four interfaces.

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^{* 3}WEAVE is a registered trademark of 3TEX Inc., Rutherfordton, NC.

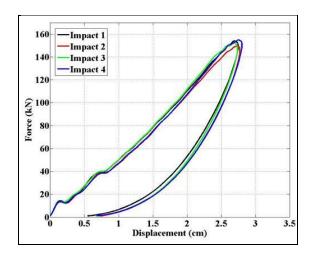


Figure 5. Force versus displacement for 3WEAVE S2/SC-15 + 10 mil TPU.

The force-deflection curves for materials incorporating TPU film inter-layers are given in figures 4–5. The material of figure 4 incorporates 5-mil TPU inter-layers and exhibits a lower degradation rate compared to the 3WEAVE S2/SC-15 material shown in figure 5. Peak loads for impacts 1–3 are higher with less impact energy absorbed by the material. The fourth impact causes more damage as evidenced by a lower peak load and increased sample deflection; however, the inclusion of 5-mil TPU inter-layers did reduce overall panel deflections from 3 to 2.5 cm compared with the 3WEAVE S2/SC-15 baseline.

Figure 5 shows that the 3-D S2 material with 10-mil TPU inter-layers exhibits the best damage resistance of the materials tested in this report. Specifically, no discernible degradation is observed between impacts and panel deflections are the lowest. This result indicates that the 10 mil interlayers are of sufficient thickness to elicit a significantly different mode of mechanical response compared to that of the other materials. This mode of response is one where delamination is essentially eliminated (discussed in section 3.2) and the panel deforms as a monolithic structure.

Figure 6 is a column chart comparing the peak tup deflections for all the materials. It is important to note, though, that these values are for comparison only since tup deflections do not account for localized crushing under the impactor and are therefore always greater than the actual back-face deflections that may be measured using optical techniques. Figure 6 shows that the 3WEAVE S2/SC-15 with 10-mil TPU exhibits the lowest deflections of all the materials tested.

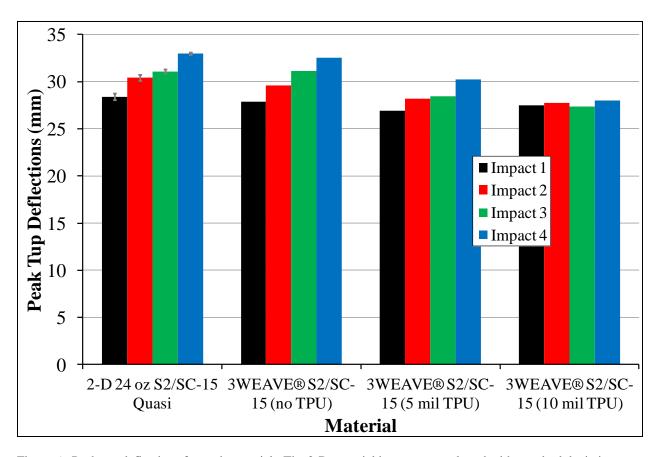


Figure 6. Peak tup deflections for each material. The 2-D material has averages plotted with standard deviation as error bars.

3.2 Damage Evolution – Ultrasonic Scans

Ultrasonic c-scan images for all the materials are presented in figures 7–10. Figures 7 and 8 show that the materials without interlayers sustain extensive delamination covering the entire aperture of the test fixture after the fourth impact. It is important to note from a comparison of figures 7 and 8 that the 3WEAVE S2/SC-15 material has a similar damage progression per impact to the baseline 2-D S2/SC-15 even though the 3-D material reinforcement only has four interlaminar regions.

Figures 9 and 10 show delamination progressions for the materials containing TPU film. The inclusion of 5-mil TPU (figure 9) only modestly improves the delamination response of the 3-D S2 panel. The delamination response of the panel with a 10-mil TPU film is given in figure 10, showing damage only in the immediate vicinity of the impact. After all four impacts, the damage is localized and the sample largely intact. The delamination areas for all materials are summarized in a column chart in figure 11.

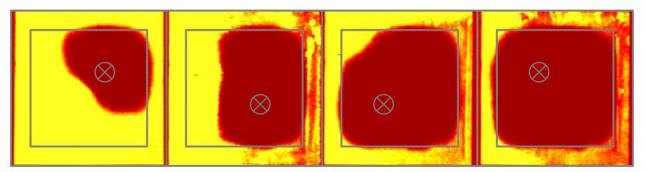


Figure 7. C-scan image showing progression of delamination in a 2-D S2/SC-15 quasi-isotropic sample.

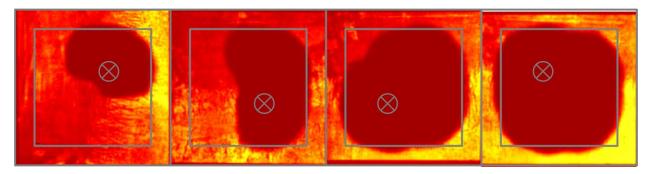


Figure 8. C-scan image showing progression of delamination in a 3WEAVE S2/SC-15 (no TPU) sample.

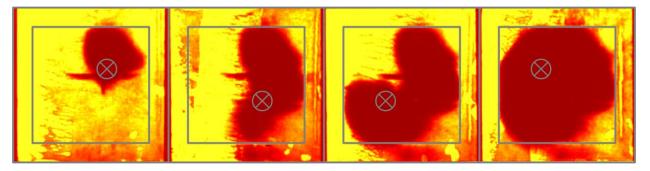


Figure 9. C-scan image showing progression of delamination in a 3WEAVE S2/SC-15 sample containing 5-mil TPU inter-layers.

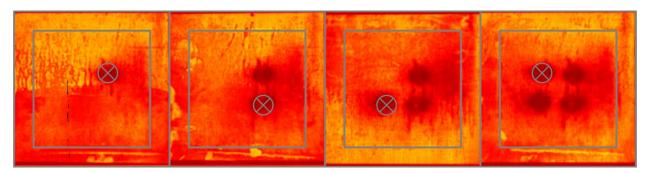


Figure 10. C-scan image showing progression of delamination in a 3WEAVE S2/SC-15 sample containing 10-mil TPU inter-layers.

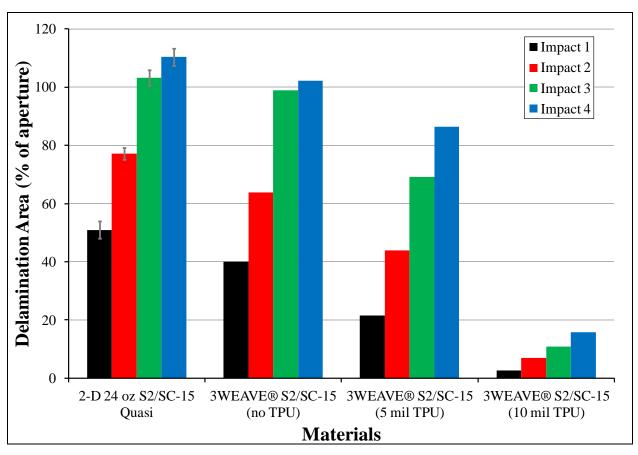


Figure 11. Delamination areas as a percentage of impact table aperture for each material. Averages are plotted for the 2-D S2/SC-15 material with standard deviations as error bars.

3.3 Failure Mechanisms – Visual Inspection

Representative images of front and back face impact damage are shown in figures 12–15. The quasi-isotropic 2-D S2/SC-15 material (see figure 12) has a characteristic pattern of damage consisting of front-face crushing immediately under the impactor, back-face perforation, and modest delamination in the vicinity of the impact. This type of damage is associated with localized punching or shear deformation. The damage progression eventually renders the sample more compliant and a transition in damage mechanisms is observed as excessive bending, out-of-plane deflection, and permanent sample deflection. After the second or third impact, the shift in the damage mechanism causes the back-face perforation to diminish or disappear (see figure 12b for the third and fourth impacts).

The 3-D reinforced materials of figures 13–15 have a different reinforcing architecture (unidirectional, non-undulating warp and weft fibers) and thus different modes of damage. Visual inspection of figure 14a and figure 15a reveal lines of localized fiber micro-buckling due to the impact and localized crushing under the impactor. The crushing is reduced compared with the quasi-isotropic 2-D S2/SC-15 material as is the back-face perforation. Predominantly what is observed in the 3-D material samples is a front-face indent and raised back-face permanent

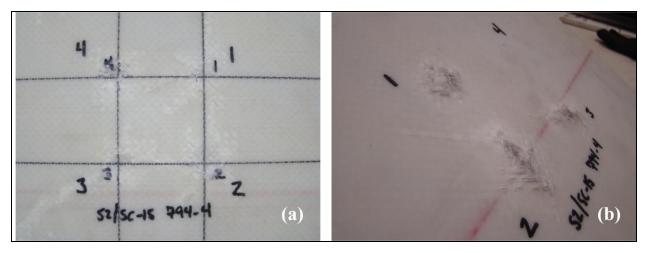


Figure 12. Front-face (a) and back-face (b) impact damage for a quasi-isotropic 2-D S2/SC-15 sample.

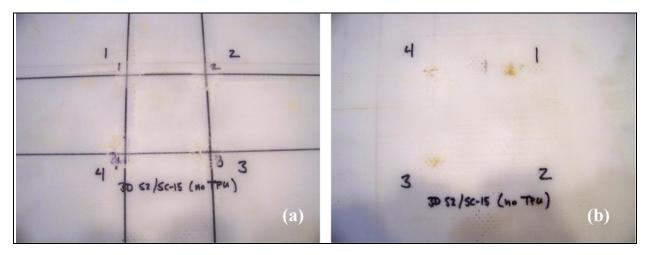


Figure 13. Front-face (a) and back-face (b) impact damage for a 3WEAVE S2/SC-15 baseline (no TPU).

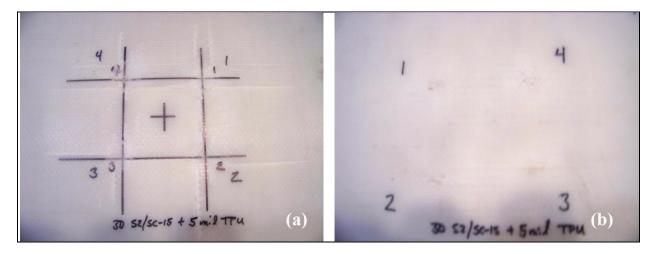


Figure 14. Front-face (a) and back-face (b) impact damage for a 3WEAVE S2/SC-15 + 5 mil TPU sample.

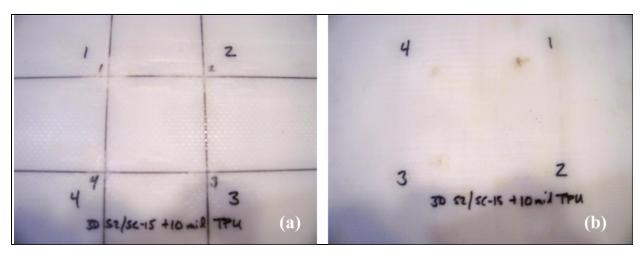


Figure 15. Front-face (a) and back-face (b) impact damage for a 3WEAVE S2/SC-15 + 10-mil TPU sample.

deflection with no or little fiber perforation. Combining information obtained from the visual images (figures 13–15) with the c-scan images (figures 8–10) for the 3-D S2-glass materials, the presumed mode of damage is localized delamination at the interfaces of the 3-D reinforcement pre-forms. The localized punching and back-face perforation so distinct in the 2-D S2/SC-15 materials is arrested by the through thickness (z-direction warp weaver) reinforcement in the pre-forms. The best performing material, the 10-mil TPU 3WEAVE S2/SC-15 (see figure 15), exhibits little external evidence that the sample was even impacted (reference c-scan images [figure 10]). The materials with TPU inter-layers then limit the delamination observed in the c-scan images to the vicinity of the impact by toughening the interfaces.

3.4 Degradation Rate

In our approach we assume that the extent of damage within the material is related to the sample stiffness (slope of the force-deflection curve) during impact. For purposes of quantification, we consider the stiffness at 1-cm deflection because the force-deflection curves (see figures 2–5) are still nominally linear up to that point and most of the transient vibration has dampened.

Figure 16 shows a plot of the initial stiffness per impact for all materials. All materials tested that did not contain any TPU film inter-layers displayed a significant drop in sample stiffness (increased rate of degradation) per impact regardless of 2-D or 3-D reinforcements. The drop was most significant after the first and third impacts. It is interesting to note again that there is little difference between the degradations of the 2-D quasi-isotropic S2/SC-15 material and the 3WEAVE S2/SC-15 material even though the 3-D material has only four interfaces and a percentage of through-thickness reinforcement.

All materials with TPU film inter-layers had higher sample stiffness and stiffness retention after successive impacts. The 3WEAVE S2/SC-15 with the 10-mil TPU film inter-layers performed best with an almost immeasurable degradation for an impact energy of 2.1 kJ. It is evident that a

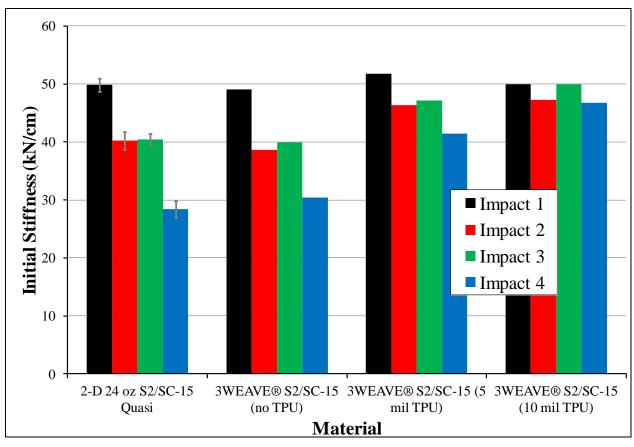


Figure 16. Initial impact stiffness values for all materials with mixed boundary conditions. Averages plotted for 2-D S2/SC-15 material with standard deviation as error bars.

decoupling mechanism was triggered upon doubling of the TPU film inter-layer thickness produced a much more durable material. This suggests that there is a threshold film thickness above which impact degradation can be significantly mitigated.

3.5 Comparison of Multi-Impact Durability

In an attempt to quantify our measurements, the present investigation uses an Ashby-type chart to compare the relative durability between the different materials under multi-impact Army relevant applications. We define the degradation rate, d_s , as the slope of the linear fit to the initial stiffness versus impact, with units of kN/cm·impact. Degradation rate, d_s , is plotted on the vertical axis and initial sample stiffness (normalized by material density) is plotted on the horizontal axis (units of kN·cm²/g). Such a chart quickly distinguishes the relative durability between different materials. Specifically, materials that appear low and to the right on the chart are more desirable from a durability standpoint than materials that plot high and to the left.

Figure 17 displays the Ashby-type chart developed in (5) to more easily assess the multi-hit durability of composite materials. The plot contains all the materials tested in this study and provides a good means of comparison between 2-D and 3-D materials with and without TPU film inter-layers. The materials without any interface toughening TPU film inter-layers are less durable and clustered near to the upper right of the plot with higher degradation rates. The best performing material has the lowest degradation rate, the 3WEAVE S2/SC-15 with 10-mil TPU.

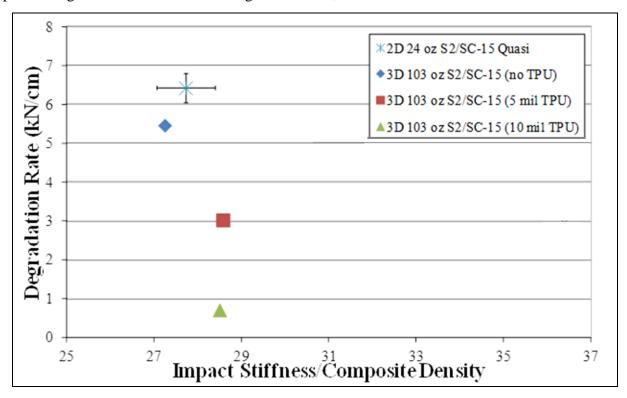


Figure 17. Ashby-type plot showing the relative durability of 2-D and 3-D S2/SC-15 composites with and without TPU film inter-layers.

4. Conclusions

Research was conducted using a newly developed LVI testing protocol to evaluate multi-impact performance of thick-section 3WEAVE S2/SC-15 composites with and without interface toughening TPU film inter-layers of varying thicknesses. The results were compared directly with impact data obtained for legacy 2-D S2/SC-15 composites with a quasi-isotropic lay-up. As mentioned, 2-D S2/SC-15 composites undergo excessive delamination damage under impact loading and exhibit significantly diminished residual properties and strengths.

Results indicate that there is very little performance gain to using stacked 3-D S2-glass pre-forms over 2-D S2-glass fabric to reinforce thick-section composites with no interface toughening. However, when TPU film inter-layers are used with 3-D pre-forms, a significant increase in durability and damage tolerance is demonstrated. The materials with 10-mil TPU film interlayers performed best having lower damage progression and degradation rates.

The main motivation of this research is to mitigate the initiation and progression of delamination through toughening of the interfaces between reinforcing fiber layers. Much work is still to be done in the following areas. Many different types of TPU film are commercially available and their unique properties need to be examined to find an optimal configuration. A modeling framework must be developed to fully investigate the significantly improved stiffness, degradation, and damage progression observed with TPU films. A hybrid composite should be developed that acquires the damage tolerance and durability afforded by the TPU film interlayers, and utilizes a synergy between glass and carbon fibers in a more balanced, hybridized pre-form. Finally, higher energy impact testing on thicker section (>25-mm-thick) composite panels must be performed and a scaled comparison conducted with the 1/2-in panels presented in this study. Such efforts are already underway at both ARL and the CCM at the University of Delaware.

The present investigation demonstrates much promise for the design of durable novel materials. The significant improvement in material durability and damage tolerance with toughened interlayers was clearly demonstrated in this work. Further development of these materials will provide the Army with more durable composite systems necessary to meet emerging structural composite applications.

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List of Symbols, Abbreviations, and Acronyms

ARL U.S. Army Research Laboratory

CCM The Center for Composite Materials at the University of Delaware

TPU Thermoplastic polyurethane

VARTM Vacuum assisted resin transfer molding

2-D Two-dimensional

3-D Three-dimensional

LVI Low velocity impact

 d_s Degradation rate

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